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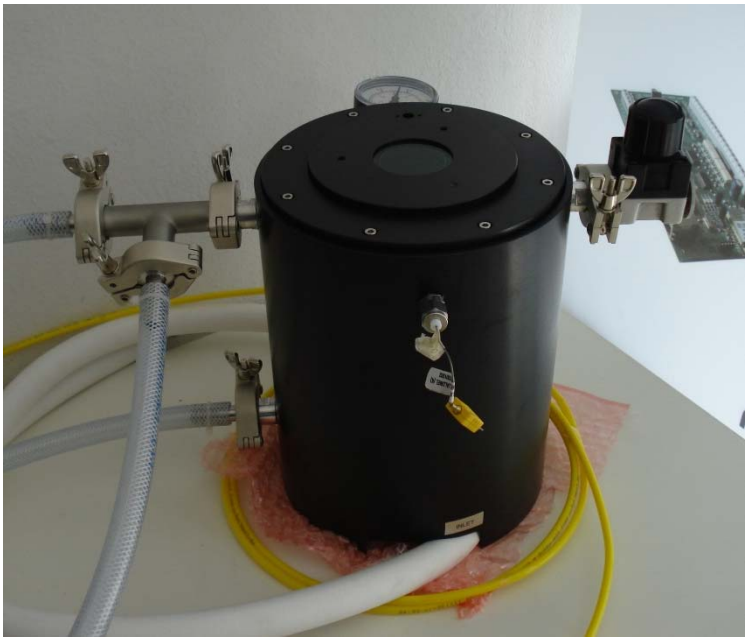
scientific electronics

TL - cryogenic oven
USER MANUAL

Rel. 01.00.0001
(Hardware code: TL-CRY)



CONCEIVING
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WARNING!
ELECTRICAL DEVICES COULD DAMAGE EQUIPMENT OR PROPERTY OR CAUSE PERSONAL INJURY

This guide contains instructions and technical features of the TL - cryogenic oven.
Read with attention before attempting to install.
It is the responsibility of the technician to undertake all the safety rules provided by the law during the installation and the use of this device.
For any information which is not contained in this guide, please contact:

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TABLE OF CONTENTS

REVISION HISTORY 6
1. DESCRIPTION 7
2. CRYOGENIC VACUUM OVERVIEW 8
3. CONNECTION OF CRYOGENIC OVEN WITH TL2000 THERMOREGULATION AND PHOTON DETECTION UNIT 12
4. CONNECTION AND INSTALLATION OF THE SYSTEM 20
5. TECHNICAL FEATURES 26
6. PARAMETER SETUP 27
7. OPTIMIZATION OF THE P-I-D CONTROL LOOP 27
8. MAINTENANCE 28
9. TECHNICAL SPECIFICATIONS 28
CONTACTS 29
SUPPORT INFORMATION 30
PROBLEM REPORT 30
ENGINEERING PROBLEM REPORT 31

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1. DESCRIPTION

The TL cryogenic oven is conceived and built to perform thermoluminescence analyses using photon detection unit and thermoregulation unit of IPSES TL2000 laboratory.

The cryogenic vacuum oven is designed to cold and heat the samples during the thermoluminescence analyses in a controlled atmosphere.
Cooling is achieved through liquid nitrogen: a copper tank is inserted inside the oven and it is filled with liquid nitrogen. Two thermal elements allow to make cold a thermal bar that supports the sample during the measurement. The thermal insulation of the copper tank filled with liquid nitrogen is achieved through vacuum.
The thermal bar on which the sample is placed is inside a camera in the oven where the atmosphere is controlled by using a vacuum root pump to remove the air and replace it with pure gaseous nitrogen from a supply line.

To achieved the cryogenic temperature the oven must be connected to liquid nitrogen under pressure cylinder: a temperature on the sample of about -140°C will be reached in about 30 minutes, starting form environmental temperature and with an empty copper tank. Once the cryogenic temperature is reached the first time and the copper tank for liquid nitrogen is full, even after an heating cycle on the sample at +300°C, the temperature of -140°C for a new analysis is reached in few minutes. To reach a lower temperature on the sample area, the oven can be connected to liquid helium, because the oven can work till -270°C. In this case a temperature of about -200°C in the sample area can be reached[2].
The thermal bar allows also to heat the sample up to +500°C.

[2] To use the oven with liquid helium for the inlet and the outlet a proper pipe not provided with the system must be used. The pipes provided with the system are suitable only for the use with liquid nitrogen

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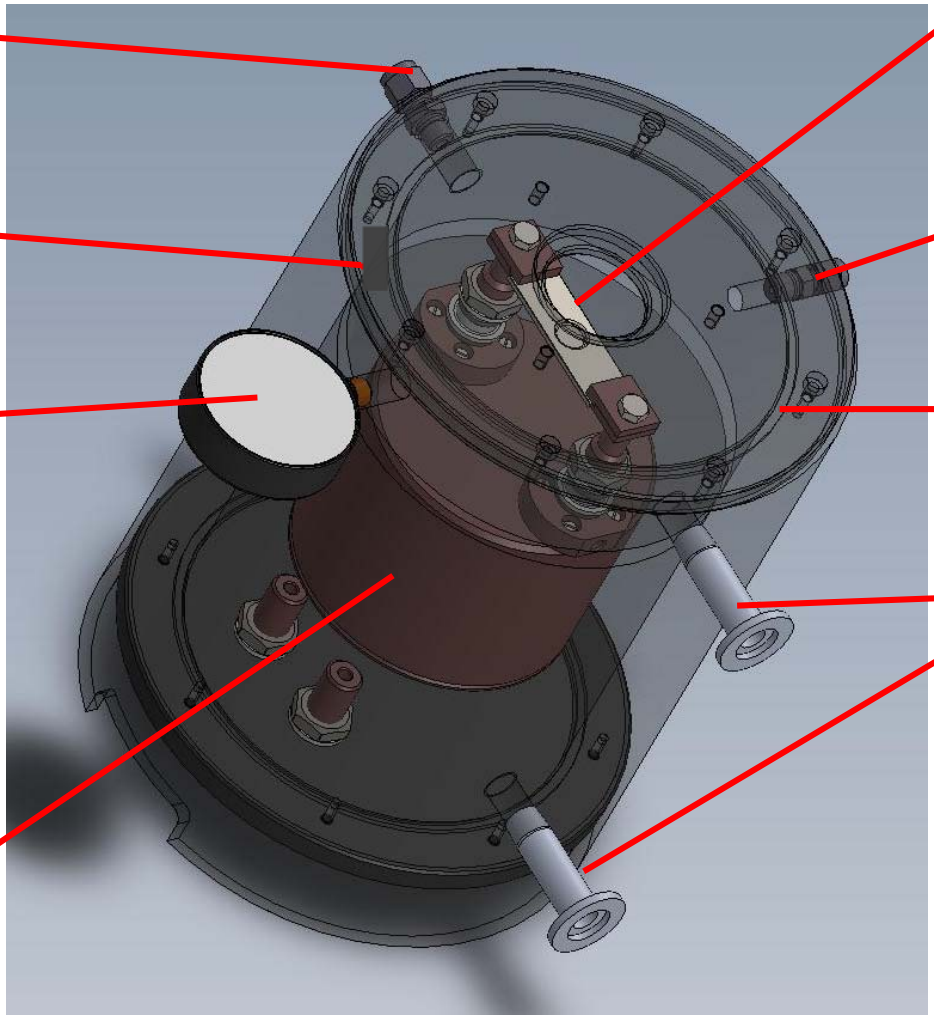
2. CRYOGENIC VACUUM OVERVIEW

The following images show the vacuum oven parts. The cryogenic oven has been conceived for been fully compatible with the photon counting unit of the vacuum oven of TL2000 thermoluminescence laboratory, so it is possible to use the same photon counting unit with both ovens, simply positioning it on the relevant o-ring.



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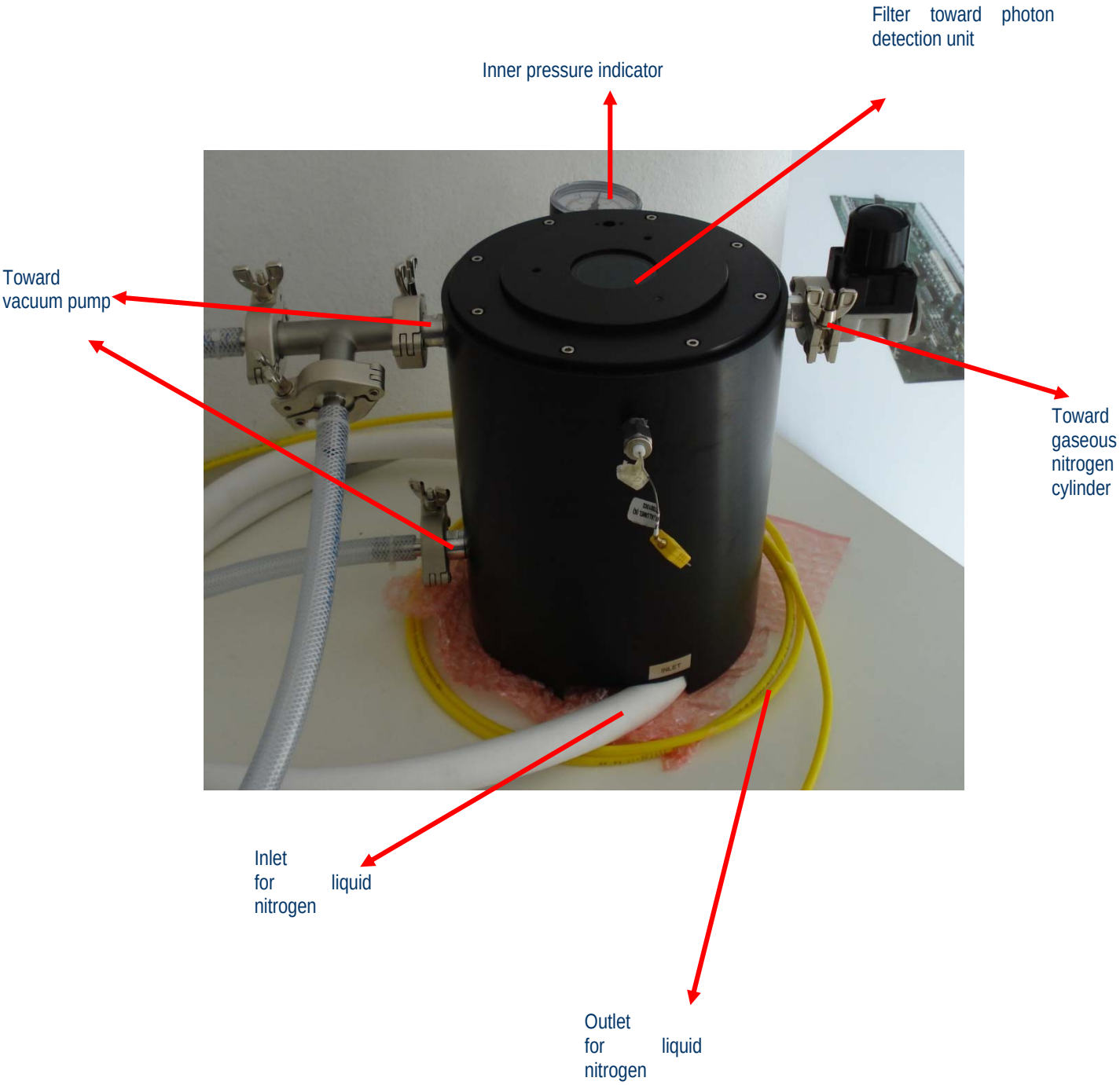

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O-ring for the positioning of photon counting unit
Inner pressure indicator
Connector to vacuum pump
Connector to gaseous nitrogen supply line
Thermocouple connector

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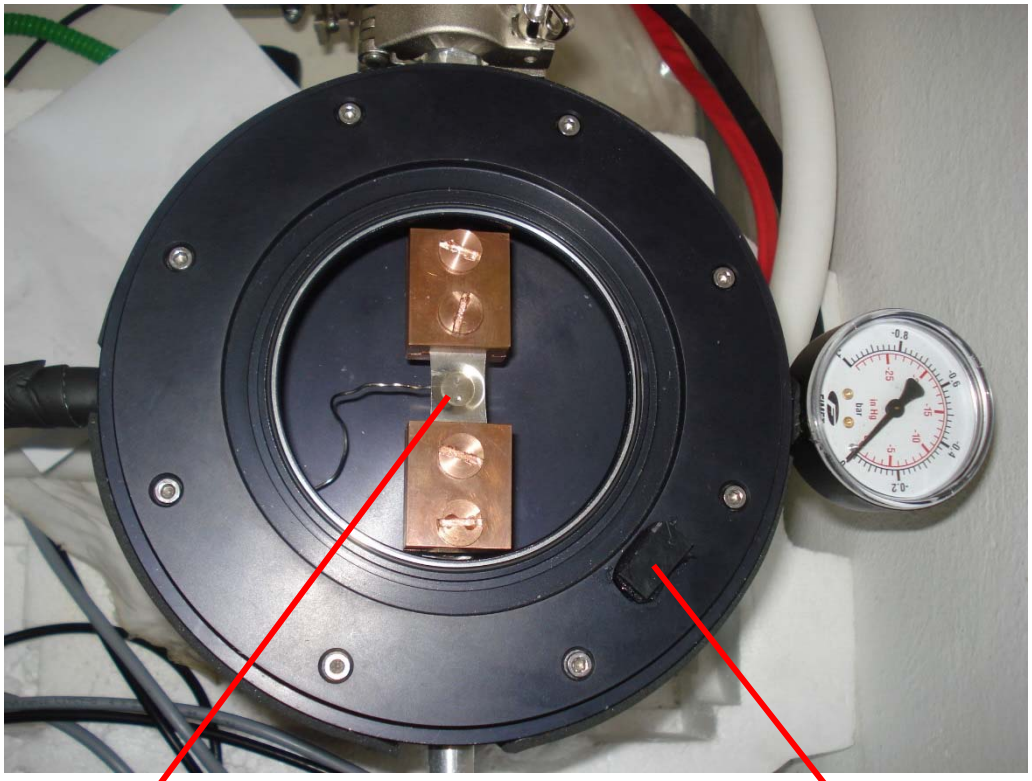


Thermal bar with sample holder

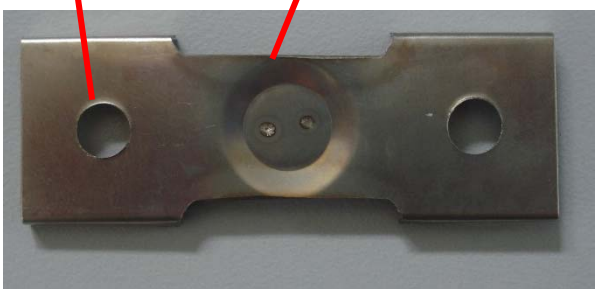
Support for switch interlock of the photon detection unit

Holes for copper screws

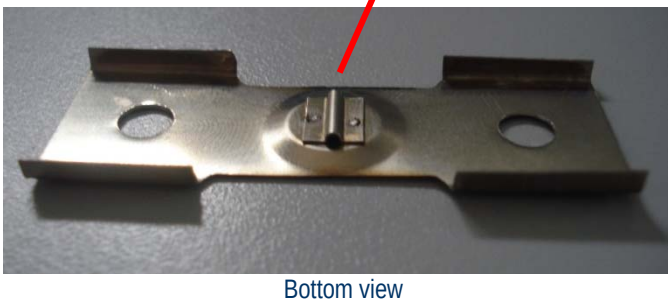
Sample holder

Socket for the thermocouple

Thermal bar



Top view



Bottom view

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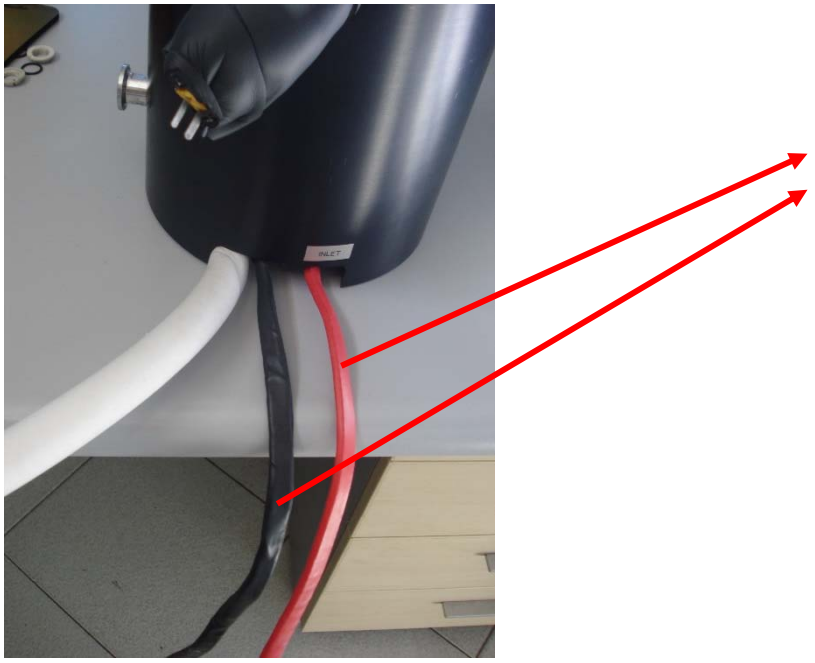
3. CONNECTION OF CRYOGENIC OVEN WITH TL2000 THERMOREGULATION AND PHOTON DETECTION UNIT

The cryogenic oven is conceived to be compatible and used with thermoregulation unit of IPSES TL2000 laboratory and photon detection unit.

TL 200O THERMOREGULATION UNIT

To connect the cryogenic with thermoregulation unit, proceed as follow:

On the base of the cryogenic oven two cables (red and black) are placed, as shown in the following picture:



Cables toward thermoregulation unit

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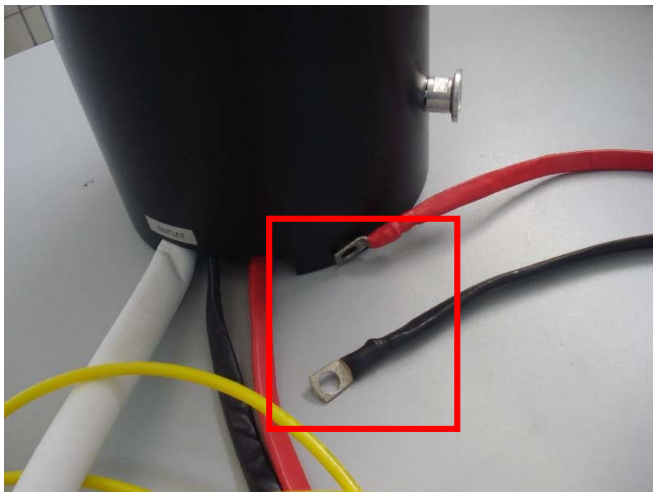
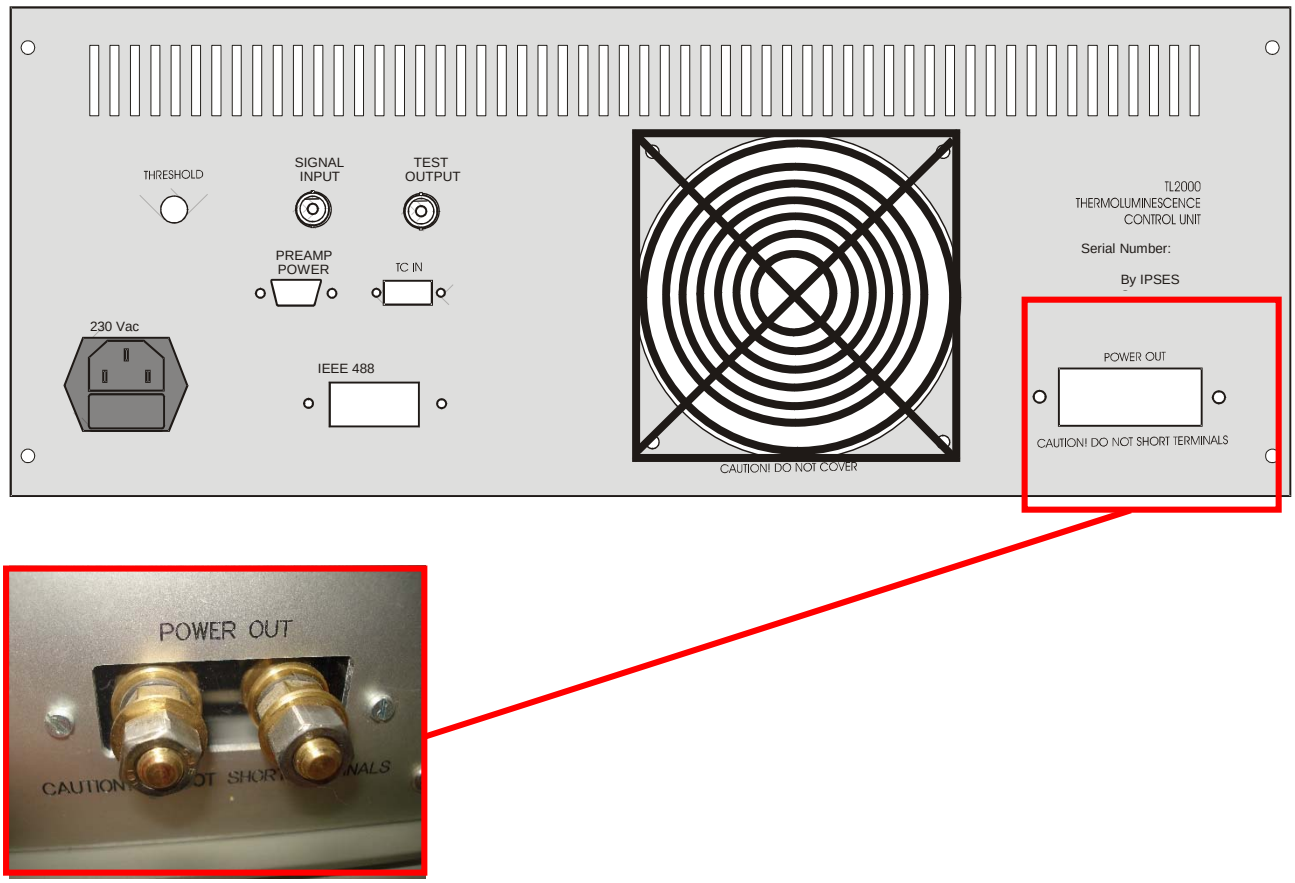
The two cables have at the end two crimp terminals, how highlighted in the following picture:



Connect the two crimp terminals on the rear of the Thermoregulation unit in the area labeled as “power out”, highlighted in the following two pictures:



To connect the crimp terminals, unscrew the two hexagon-nuts on the two connectors placed on the rear of the thermoregulation unit, insert the crimp terminals, then screw the two hesagon-nuts. The position of the cables (black and red) is not mandatory: they can be connected either on the right and on the left.

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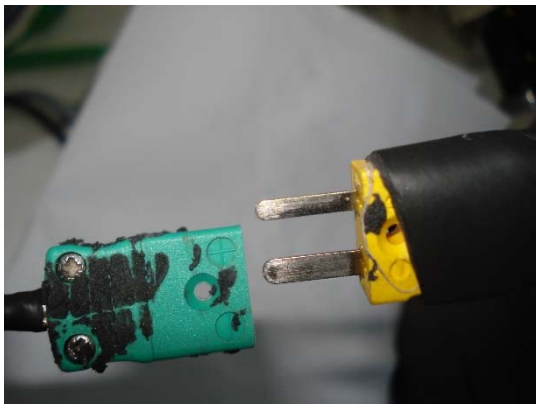
Then connect the thermocouple plug to the relevant wire equipped with a female connector as shown in the following pictures. Connect the plus pole and the minus pole to the relevant side (plus and minus are marked on the plugs), in any case the plug can be inserted in the female connector only in the correct side.



The thermocouple coming out the oven has been thermally insulated to reduce the heat transmission (due to environment temperature) inside the oven.

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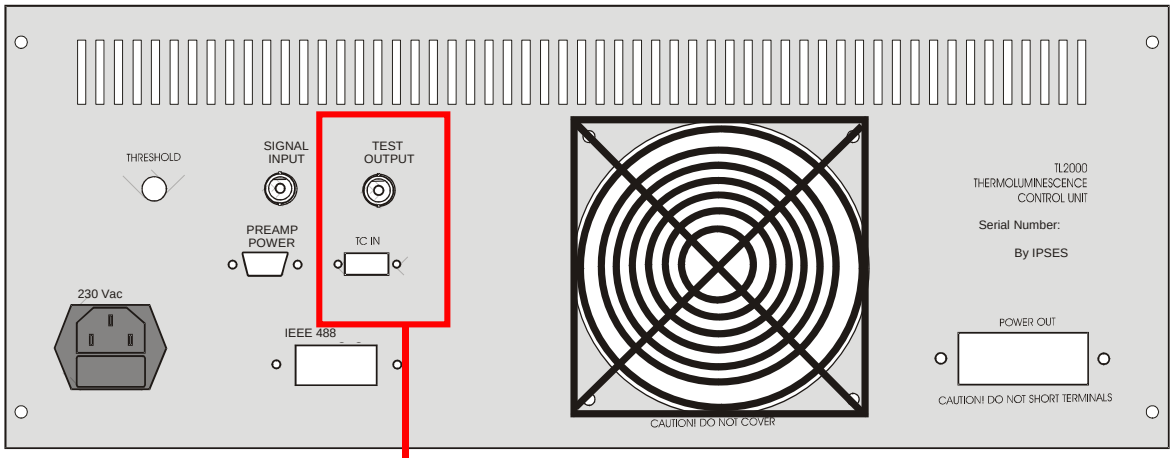
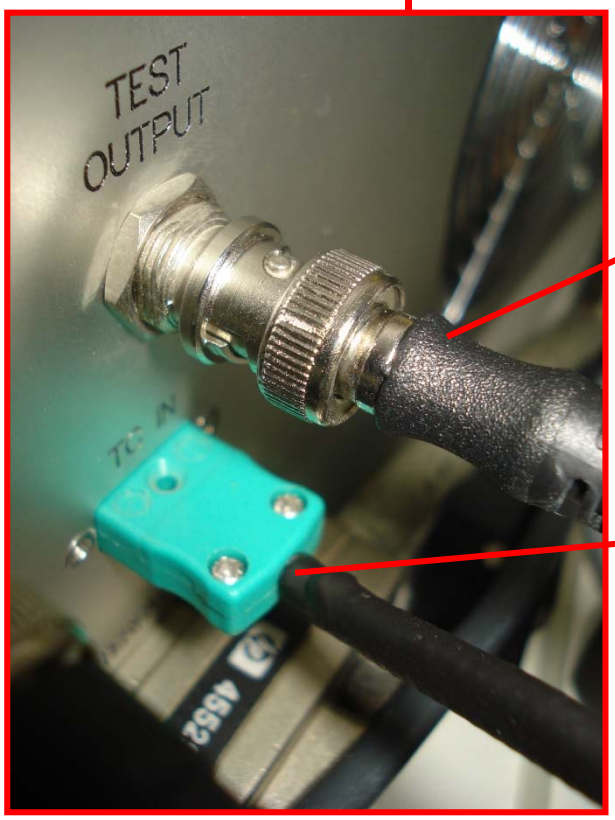
Then plug the other side of the wire connected to the thermocouple on the TL2000 thermoregulation unit, as shown in the following pictures. The wire ends with two separate connectors: a round BNC connector that must to be plug on “TEST OUTPUT” and a male TC connector that must to be plug on “TC IN”. For both connectors they can be inserted only in the correct position.





Round BNC connector

Male TC connector

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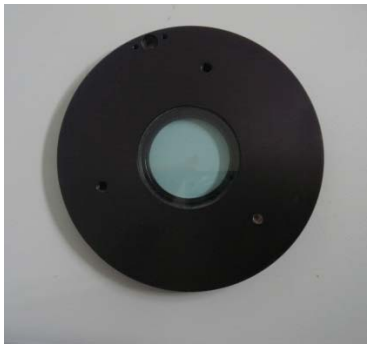
Due to the fact that a cryogenic temperature the signal coming from the thermocouple is low and can be effected by noise, the cable has been shielded.

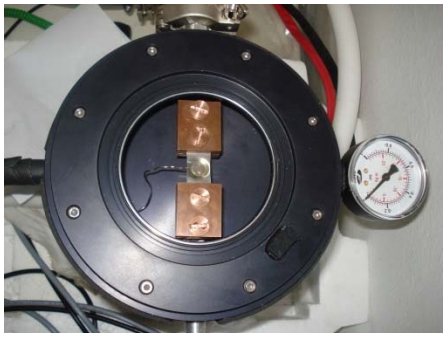
Once the oven is connected to the thermoregulation unit, the photon counting unit can be mounted on the oven.

The cryogenic oven has been designed to be compatible with the flange of the IPSES vacuum oven used for TL analysis: so it is not necessary to unscrew the photomultiplier housing form that flange and mount it on the new flange of the cryogenic oven. You can just take off the extra flange from the cryogenic oven and simply positioning the photon counting unit on the ring, as shown in the following pictures:

Cryogenic oven equipped with its flange (top view)



Take off the flange



Cryogenic oven without flange (top view). Pay attention that the O-ring is all inside the groove

Photon detection unit mounted on the flange of the TL2000 vacuum oven

Just put the photon detection unit on the cryogenic oven

The unit will fit exactly on

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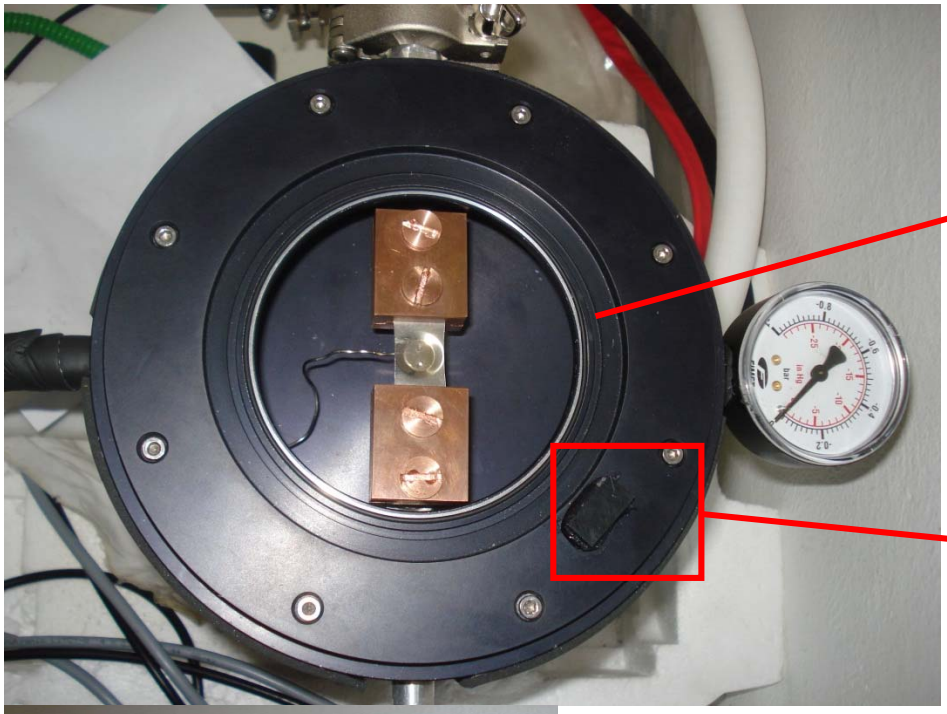
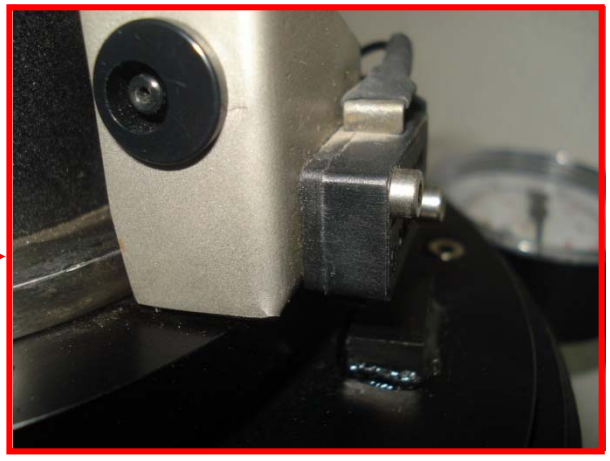
When you place the photon detection unit on the cryogenic oven, pay attention to put the interlock switch over the interlock switch support, as shown in the following pictures:





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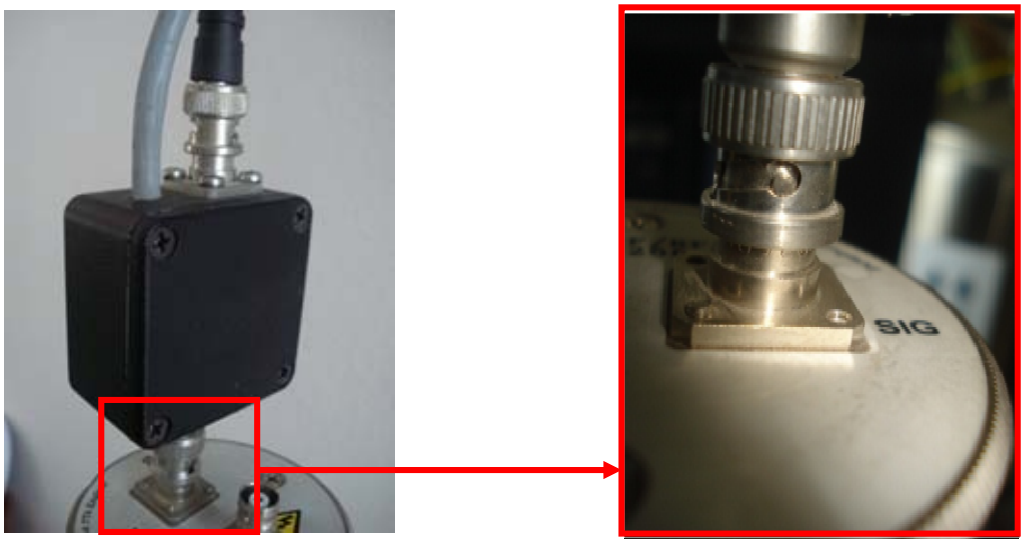
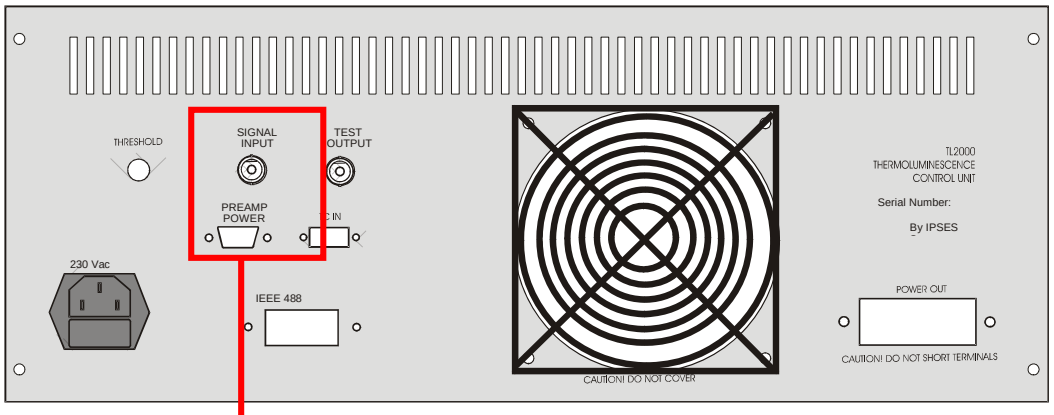
At this point the pre-amplifier Barpa can be mounted on the photon detection unit:

Insert the Barpa male connector on the female connector of the photomultiplier housing labeled with “SIG”.



Then connect the two cables of Barpa with the TL2000 thermoregulation unit, on the two connectors labeled “SIGNAL INPUT” and “PREAMP POWER”:



Detail of Barpa cables connected to the thermoregulation unit. It is possible to insert the connectors only in the right position.

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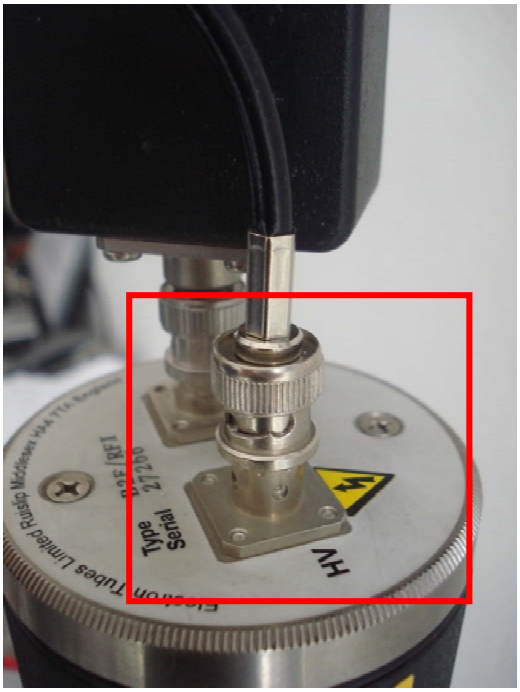
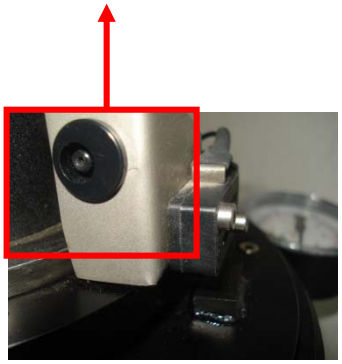
Then connect the High voltage power supply mod. HiVo to the photon counting unit: insert the relevant male connector on the female connector of the photomultiplier housing labeled with “HV”. Then connect the other side of the wire to the connector labeled “H.V. out” on the rear side of HiVo.



Connector for the interlock cable on the photon counting detection unit



Connector for the interlock cable on the rear side of HiVo

Then connect the interlock cable to the “Interlock H.V.” connector on the rear side of HiVo, and the interlock connector on the photon detection unit.

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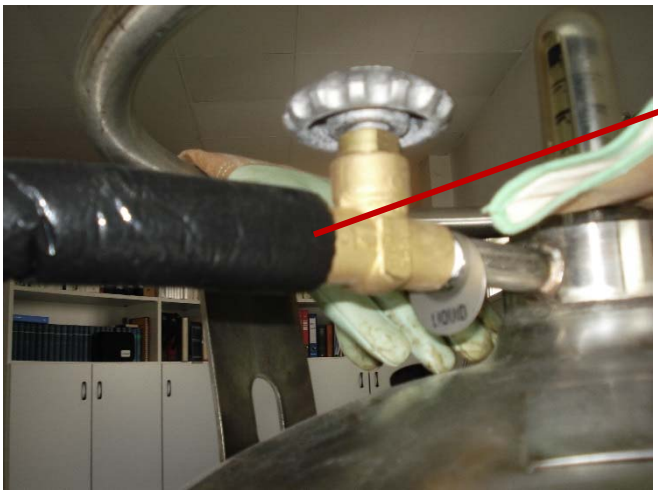
4. CONNECTION AND INSTALLATION OF THE SYSTEM

Connect the black pipe (provided with the system) to the liquid nitrogen cylinder. The cylinder must be under pressure:

Pipe toward liquid nitrogen



Detail of the pipe connected to liquid nitrogen cylinder

The connection is achieved through a screw flange at the end of the black pipe which is compatible with the most part of screw connectors of cylinders for liquid nitrogen (otherwise an adaptor is required). Once the black pipe is connected to the under pressure cylinder, connect the other side of the pipe with the oven, through the screw connector shown in the following picture:

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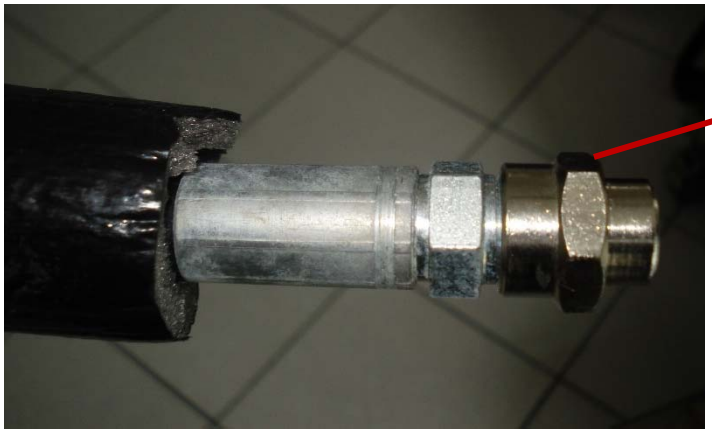
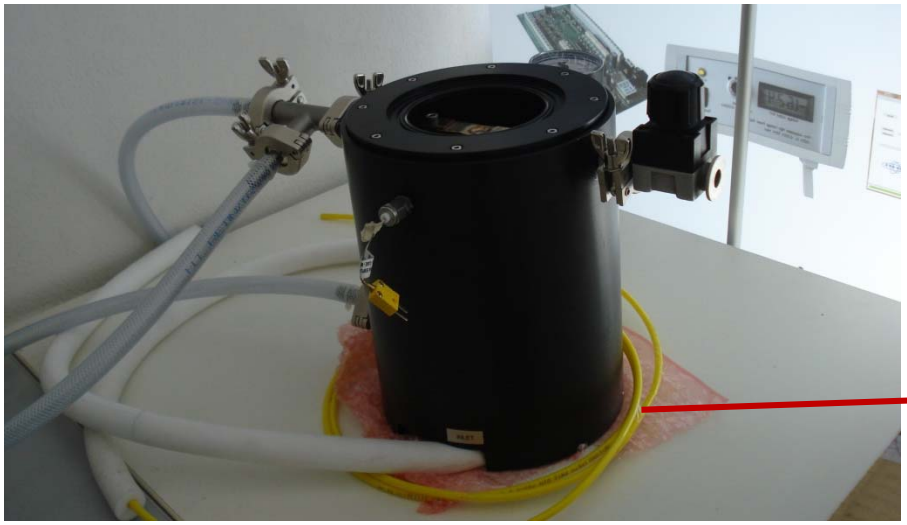


Detail of the black pipe – side with the connector toward the oven

At this point, put the yellow pipe for the nitrogen output in the Dewar provided with (pay attention: positioning the Dewar in a stable position, because during the analysis, liquid nitrogen can fill inside):



Yellow pipe for nitrogen outlet

Detail of the yellow pipe for nitrogen outlet in the Dewar

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The cryogenic vacuum oven must be connected to a vacuum pump by two connectors that are shown in the following image:

The connectors are two standard flange type DN16KF: they can be connected to a unique vacuum pump using proper connectors and speedy valves (not provided with the system). Two speedy valves are required, one for each flange.
In the opposite side of the oven there is the connector toward gaseous nitrogen, as shown in the following picture:

The connector is a standard flange type DN16KF.

The connection must be achieved using proper connectors and speedy valve to control the nitrogen flow (not provided with the system).

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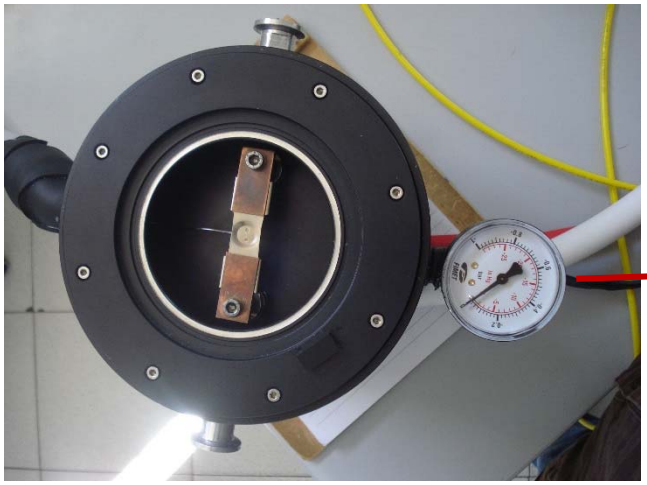
Detail of connection toward vacuum pump (upper flange) with speedy valve and clamps

Detail of connection toward the gaseous nitrogen with speedy valve and clamps

During analysis to have the correct inert atmosphere inside the camera, it is recommended to proceed as follow:

First turn on the vacuum pump and open the two speedy valves toward the oven till the inner pressure is about -1 bar (end of scale of the pressure indicator).



Inner pressure indicator

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Keep the vacuum pump on during all the analysis process. Once the pressure is near -1 bar, gently open the speedy valve toward gaseous nitrogen: by this way the nitrogen will filled the camera and the inert atmosphere is correctly reached.

Then close the nitrogen speedy valve completely, otherwise the flow of gaseous nitrogen will heat the temperature inside the chamber. Remember to keep both the valves toward the vacuum pump completely open so to have an inner pressure of about -1 bar.

At this point you can gently open the liquid nitrogen valve and leave the nitrogen going inside the cryogenic oven.

At the beginning from the outlet yellow pipe only gaseous nitrogen will come out. Once the copper tank is fulfilled, also from the yellow pipe, together with the gaseous nitrogen, the liquid one will come out.

PAY ATTENTION: nitrogen has a very low temperature and all the objects coming in contact with it, can keep a temperature that can burn skin.

Due to low temperature, around the area on which there is a passage of liquid nitrogen the environmental humidity will frost. This is normal and do not affect the working of the system, because inside the oven there is vacuum, so nothing can frost.
A high presence of environmental humidity (upper 60% Rh) anyway is not recommended.

Once the system is operative and cooled, to insert the sample in the thermal bar, act as follow:

1. Close the liquid nitrogen valve.

2. Close the speedy valve toward the vacuum pump only in the upper part of the oven, keeping open the lower one, by this way the vacuum will be kept around the copper tank filled with liquid nitrogen.

3. Open the speedy valve of the gaseous nitrogen till have not the vacuum inside. At his point the photon unit with its flange can be taken off.

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4. Close completely the speedy valve of the gaseous nitrogen.

5. Put the sample on the thermal bar. This operation must be performed as faster as possible, to avoid a too high warming of the camera by the environmental temperature.

6. Close the camera with the photon counting unit, gently open the upper speedy valve toward vacuum pump till to reach an inner pressure of about -1 bar. This operation must be performed slowly, to avoid that some of the air present in the upper camera will pass into the lower camera.

7. Gently open just a little bit the speedy valve toward gaseous nitrogen to be sure to have an inert atmosphere inside.

8. Close the speedy valve toward the gaseous nitrogen, when the indicator of the manometer reach a -0,9 or -0,8 bar.

9. Open the liquid nitrogen valve.

10. When you are sure that the inner copper tank is fulfilled of liquid nitrogen (i.e. when liquid nitrogen starts to come out form the outlet yellow pipe), it is possible to close the liquid nitrogen valve. When you see the temperature on the sample area starts to arise (without stimulation), open again the liquid nitrogen valve.

5. TECHNICAL FEATURES

Sample cooling is obtained by two thermal elements connected to a small tank filled with liquid nitrogen. Sample heating is obtained through a heating resistive element. The temperature is measured through a type-K thermocouple (heating resistive element and thermocouple are provided with the unit).

The control of the power supplied to the heating element is achieved by the use of a PID loop: the error signal, obtained as difference between the reference temperature at a given time t and the actual temperature measured by the thermocouple, is differentiated and integrated. The three components (Proportional, Integral and Differential) are

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independently amplified (the gain values can be adjusted by the user). The accuracy of the heating profile can be minimized by adjusting the gains of the PID control loop. Different installation conditions (length of the oven cables, operating pressure, dimension and type of the sample) can require an adjustment of the default gain settings.

The measure of the intensity of the light emitted by the sample during the heating (glow curve) is achieved by photon counting: a proper PM tube suitable for this operating mode (low dark current, high focusing) must be used. Optionally, the PM tube can be purchased as part of the TL2000 control system. The overall quality of the measurements performed by the system is affected by the correct choice of the PM tube.

PAY ATTENTION: the cryogenic oven is conceived to perform photon counting measure starting from a cryogenic temperature. If the cryogenic oven is used without liquid nitrogen inside to perform classical TL measure (for example starting from +50°C up to 500°C), the linearity of the heating ramp cannot be reached due to mechanical design of the thermal bar. To perform classical TL , it is recommend to use the TL2000 vacuum oven.

6. PARAMETER SETUP

Working parameters of the unit (gains of PID, parameters for the interface, maximum temperature reachable, maximum during time of the heating cycle) can be set by the user, pressing the SETUP key on the front panel of the TL unit (see the relevant user manual). Since the thermal impedance on the cryogenic oven is lower than the one of the TL2000 vacuum oven, PID parameters have to be set differently.
To set the parameters, see the relevant paragraph (parameter setup) on the TL2000 user manual that can be downloaded from IPSES website at the following address: http://www.ipses.com/PDF/IPSES-TL-user%20manual-en.pdf.

7. OPTIMIZATION OF THE P-I-D CONTROL LOOP

The adjustment of the P-I-D gains for the three components (Proportional, Integral and Differential) allows optimizing the behavior of the unit so to have a minimum error between the theoretical and the real heating profile. If the heating system (strip, cables, working atmosphere) changes or if user is analyzing samples which have a very different thermal capacity, the gain values need to be adjusted. We suggest to follow the procedure listed below:

- Set to 1 the I and D components, then increase the gain of the P component until the heating is effective up to the end temperature of the desired ramp
- Increase the D gain to minimize the fluctuations and ringing of the temperature
- Increase the I gain to minimize the temperature error
- Repeat the adjustments a few times until the results are satisfactory

Note: an I gain set too high can cause fluctuations of the temperature which have a progressive increase of excursion and period (minutes); a high D gain can cause instability of a shorter period (seconds) and an irregular period.

Here below there are the suggest setup values for the traditional vacuum oven and for the cryogenic oven:

	TL 2000 vacuum oven	Cryogenic oven (for heating ramp with rise rate of 5°C/sec and 10°C/sec)	Cryogenic oven (for heating ramp with rise rate of 15°C/sec, 10°C/sec and 5°C/sec)
P	60	35	35
I	3	3	3
D	80	80	120

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PAY ATTENTION: after every modification of PID parameters, it is necessary to switch off and then switch on the TL2000 thermoregulation unit and restart the software.

8. MAINTENANCE

Check periodically the clamping of the power supply cables of the strip, control the strip is well fixed to its terminals: a loose connection can cause a failure in power transfer and a decay of the heating ramp and can also cause damages to the electrical terminals.
Clean the optical filters with a soft cloth without hairs. Follow maintenance instructions indicated in its user manual for the vacuum pump.

9. TECHNICAL SPECIFICATIONS

1 – Power supply of the heating strip
- maximum voltage 5 V
- maximum current 90 A
- maximum power 450 W

2 – Measure of the temperature
- thermocouple type K input
- linearity +/- 1° C
- *range* from -200° C up to +500° C (centigrade degrees)

3 – Working temperature
- environment temperature: 0°C up to 40°C
- Humidity: 0 up to 60% Rh
- Inner temperature inside the oven: -260°C up to + 500° C

4- connection of the cryogenic oven
- Three flange connectors type DN16KF

Mechanical dimensions: diameter 200 mm, high (without photon counting unit) 256 mm (diameter 7,87 inches; high 10.8 inches)

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PROBLEM REPORT

The next page is a standard template used for reporting system problems. It can be copied and send as a fax. Alternative bugs may be reported by emails, in this case please insure that the mail contains similar information listed in the *Engineering Problem Report* form.

ENGINEERING PROBLEM REPORT

Problem describer

Name			**IPSES s.r.l.** **Via Suor Lazzarotto, 10** **Cesate (MI)** **Italy** **Fax (+39) 02 700403170** **e-mail *support@ipses.com***
Company			
Date	Tel.	Fax	

Product

Name	Version	Serial No.

Report Type (bug, change request or technical problem)

Major bug	☐	Urgency:	
Minor bug	☐	High	☐
Change request	☐	Medium	☐
Technical problem	☐	Low	☐

Problem Description

Reproduction of Problem

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Received by	Date	Report No.	Action

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